7.1. Summary

In conclusion, we have carefully examined the catalytic performance of mixed metal oxide catalysts, proceeding to mixed metal selenides, and further adding the conducting polymer polypyrrole to build composite materials. Through this work, we first explored the base catalysts and then assessed their functionality by doping metals into the crystal lattice. The findings suggest that these alterations lead to considerable increase in both catalytic activity and stability. The doping of metals in the crystal structure seems to boost the overall performance, indicating a possible technique for enhancing catalytic systems in future applications.

In **Chapter 1**, we offered a detailed introduction to the Oxygen Evolution Reaction (OER), covering its underlying principles, relevance in energy conversion and storage technologies, and the vital function it plays in systems such as water splitting for hydrogen synthesis. We also additionally looked into the numerous kinds of electrocatalysts utilized for OER, including transition metal oxides, hydroxides, and selenides, stressing the aspects that impact their catalytic efficiency. In particular, the chapter emphasized essential characteristics such as the Tafel slope, which is critical for assessing the performance of OER catalysts. Furthermore, a full study of the molecular pathways of OER catalysis was offered, detailing the involvement of numerous intermediates, such as hydroxyl and oxy species, and how their production and adsorption energy impact the overall reaction kinetics.

We covered in detail the many physicochemical and electrochemical characterization methods that were used to assess the catalysts that we utilized in our experiments in **Chapter 2**. Each method's importance in gaining vital information about the catalysts' structural, compositional, and electrochemical characteristics was highlighted

with a thorough explanation. The significance of certain techniques in identifying the crystal structure, morphology, and elemental composition of the catalysts was emphasized. These techniques included X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), and high-resolution transmission electron microscopy (HR-TEM). Understanding the characteristics of the material and how they affect catalytic behavior requires the use of these techniques.

The electrochemical methods of evaluating the catalytic performance, such as electrochemical impedance spectroscopy (EIS), linear sweep voltammetry (LSV), and cyclic voltammetry (CV), were also investigated. These techniques are essential for assessing important performance metrics that are essential for comprehending the stability and effectiveness of OER catalysts, such as the charge transfer resistance, Tafel slope, overpotential, and onset potential. Our ability to correlate the electrochemical performance with the physicochemical features allowed us to make significant inferences on the connection between the structure of the material and its catalytic activity.

In **Chapter 3**, we presented a straightforward and cost-effective synthesis method for the preparation of zinc ferrite ($Zn_xFe_{3-x}O_4$; $0<x\leq1$) particles, utilizing egg white as a precursor. By varying the stoichiometries of zinc (Zn) doping, we investigated the influence of zinc incorporation on the catalytic properties of the resulting materials. This approach not only offers an economical alternative to traditional synthesis methods but also facilitates the fine-tuning of the material's composition for enhanced catalytic performance. The synthesized Zn ferrite catalysts exhibited promising electrocatalytic activity demonstrating improved efficiency. The catalytic performance was evaluated in terms of key parameters, showing that Zn doping significantly enhanced the material's ability to drive the OER with lower energy requirements. Furthermore, the catalysts displayed

excellent long-term stability, maintaining high activity over extended periods, which is critical for practical applications in energy conversion devices. zinc ferrite particles exhibit remarkable multifunctionality, making them promising candidates not only as efficient catalysts for the oxygen evolution reaction (OER) but also for broader applications in energy storage and conversion technologies. Their unique structural, electronic, and redox properties enable them to participate in fast charge-transfer processes, which are critical for both catalytic activity and energy storage performance. Beyond OER, zinc ferrite has shown potential in applications such as lithium-ion batteries, supercapacitors, and photocatalysis, highlighting its versatility and value in advancing sustainable energy solutions.

In **Chapter 4**, manganese selenide (α-MnSe) and polypyrrole (ppy) heterostructure was synthesized, characterized, and its electrochemical performance as an electrocatalyst for the OER were investigated. In order to demonstrate the promising catalytic performance of the α-MnSe/ppy composite, the effort attempted to construct a highly efficient, non-noble metal-based catalyst that could achieve a current density of 10 mA cm^{-2} with an extraordinarily low excessive potential of 168 mV (vs. RHE). We used *operando* UV-Vis spectro-electrochemical methods to monitor the formation of intermediate species during the OER in order to get a better understanding of the catalytic process. This method yielded important insights into the mechanics of the reaction and the catalyst's function in promoting effective oxygen evolution.

According to the study's findings, the manganese selenide-polypyrrole composite has the potential to be a very effective electrocatalyst for OER, performing on par with or better than conventional benchmark catalysts made of manganese and rare-earth metals.

In **Chapter 5**, we investigated the effect of doping molybdenum (Mo) into the manganese selenide (MnSe) lattice to enhance its electrocatalytic activity for the OER. Additionally, to further improve the catalytic performance, we synthesized a composite of Mo-doped $MnSe_2$ with the conducting polymer polypyrrole (ppy). The incorporation of Mo into the lattice was found to significantly influence the electronic structure of the material, leading to enhanced catalytic properties. The Mo-doped α-MnSe/ppy composite exhibited a quite low overpotential of 205 mV (vs. RHE) at a current density of 10 mA cm^{-2}. The presence of Mo likely facilitates the activation of the catalyst surface and improves charge transfer, thereby lowering the energy required for the OER.

Overall, the findings provide a clear demonstration of how doping strategies and the incorporation of conducting polymers can be utilized to tune the properties of metal chalcogenides, enhancing their catalytic performance for renewable energy applications.

In **Chapter 6**, we investigated the effect of copper (Cu) doping on the catalytic activity of manganese selenide for the OER. The Cu-doped MnSe catalyst showed enhanced catalytic performance compared to its undoped counterpart, although it exhibited a higher overpotential of 299 mV (vs. RHE) to achieve a current density of 10 mA cm^{-2}, indicating that it did not outperform the Mo-doped variant. *Operando* spectro-electrochemical studies were conducted to examine the active intermediates formed during the OER process, providing valuable insights into the mechanistic differences between Cu doping and other doping strategies. These findings underscore the potential of metal doping, including Cu, in tuning the catalytic properties of non-noble metal electrocatalysts for OER applications.

Overall, this chapter contributes to a deeper understanding of doping strategies and their impact on the electrochemical behavior of MnSe-based catalysts, advancing the

development of efficient and cost-effective electrocatalysts for sustainable energy technologies.

7.2. Future Scope and Perspective

The results of this thesis provide a number of exciting directions for further investigation and advancement in the area of Oxygen Evolution Reaction (OER). Future research should concentrate on investigating other doping techniques, enhancing catalyst stability, and developing a better understanding of the mechanistic elements affecting OER performance. The practical use of these materials will also depend on scaling up the manufacturing and incorporating these catalysts into actual energy devices like fuel cells and electrolyzers for water splitting. We can get closer to creating affordable, effective, and long-lasting electrocatalysts for sustainable energy systems by tackling these obstacles.

1. By Improving the synthesis methods to get exact control over the composition, shape, and uniformity of doping in catalysts and creating atomic-level doping procedures to enhance catalysts' electrochemical characteristics.
2. To enhance the conductivity, stability, and general electrochemical performance of the catalysts, we could investigate the use of conducting polymers other than polypyrrole (ppy).
3. Further investigation is needed to understand the active intermediates and reaction processes using *operando* spectro-electrochemical methods to get a better understanding of the variables influencing catalytic performance.
4. We must ensure these catalysts satisfy the requirements of commercial applications by refining them for usage in extensive renewable energy systems.

5. By examining the catalysts' long-term stability and performance under realistic operating circumstances to guarantee their dependability and effectiveness for sustainable energy technologies.

The design of catalysts, their use in electrochemical energy conversion processes have all been better understood in this thesis. Additionally, it has brought attention to how critical it is to comprehend the structure-activity link among catalysts in order to build highly efficient catalyst systems.

The overall findings of this thesis highlight the pivotal role of metal doping in tailoring the structural, electronic, and catalytic properties of manganese-based electrocatalysts for the oxygen evolution reaction (OER). Through systematic investigation of various dopants, including zinc (Zn), molybdenum (Mo), and copper (Cu), we demonstrated that doping serves as an effective strategy to modulate the electronic environment and improve charge-transfer kinetics, ultimately enhancing catalytic performance. Among the studied dopants, Mo incorporation into the MnSe lattice yielded the most promising results, achieving a low overpotential of 205 mV at 10 mA cm^{-2}, likely due to its ability to activate surface sites and facilitate electron mobility. Zn doping in zinc ferrite ($Zn_xFe_{3-x}O_4$) also showed significant improvements in catalytic efficiency and stability, while Cu doping introduced additional active sites, albeit with slightly higher energy requirements. The combination of doping with conducting polymers such as polypyrrole (ppy) further amplified these effects by promoting better charge transport and active site exposure. Collectively, the results affirm that rational doping strategies, coupled with hybrid material design, provide a powerful approach to develop highly efficient, earth-abundant electrocatalysts for sustainable energy conversion technologies.